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respectively. With these amendments, it is submitted that the rejection over the combination of Carpenter and Kratel should be withdrawn.

The rejection of claims 1, 4-6, 23, and 27-32 under 35 U.S.C. § 103 for obviousness over Japanese Kokai Patent Application No. 138145 to Kawaguchi et. al. ("Kawaguchi") in view of European Patent No. 38,900 to Schwarz et. al. ("Schwarz") is respectfully traversed.

Kawaguchi relates to a method of making a quartz glass element by oxidizing a silane compound or a siloxane compound (e.g., hexamethyldisiloxane). There is no disclosure of reacting a polymethylcyclsiloxane.

Schwarz manufactures pyrogenic silica with siloxanes, such as hexamethylcyclotrisiloxane or octamethylcyclotetrasiloxane.

Applicants submit that the combination of Kawaguchi and Schwarz is improper, because these references are directed to making products which are chemically and physically completely different from one another.

On one hand, Kawaguchi is making quartz glass elements to be used in a lens for a photolithography device. Chemically, the quartz glass is silicon dioxide ( $\text{SiO}_2$ ). On the other hand, Schwarz is directed to making pyrogenic silicic acid, also known as fume silica or pyrogenic silica. As demonstrated in J. Grant, Hackh's Chemical Dictionary p. 610 (1969)(attached hereto at Exhibit 1), silicic acid has the formulae  $\text{H}_2\text{Si}_4\text{O}_9$ ,  $\text{H}_2\text{Si}_2\text{O}_5$ ,  $\text{H}_4\text{Si}_3\text{O}_8$ ,  $\text{H}_2\text{SiO}_3$ ,  $\text{H}_8\text{Si}_3\text{O}_{10}$ ,  $\text{H}_6\text{Si}_2\text{O}_7$ , or  $\text{H}_4\text{SiO}_4$  and, therefore, it is chemically very different from silicon dioxide quartz glass. This is emphasized by Kirk-Othmer Encyclopedia of Chemical Technology, Vol. 21, pp. 993 and 1009 (4<sup>th</sup> ed. 1997) (attached hereto at Exhibit 2) which teaches that fumed or pyrogenic silica can be made from (and, therefore, must be different from) silicon dioxide. Further, Schwarz's pyrogenic silicic acid is also physically distinguishable from the solid glass components of Kawaguchi which are used in lenses. As set forth in Schwarz, pyrogenic silicic acids are highly dispersed (i.e. finely divided) high surface area particles. These particles are useful as thickeners in toothpaste, varnish, and paint, and are produced by a completely different process than that of Kawaguchi. Specifically, Kawaguchi utilizes soot deposition to make a glass body, while Schwarz avoids deposition in order to produce dispersed particles. Since the silicon dioxide of Kawaguchi is physically and chemically so very different from silicic acid, one of ordinary skill in the art would have had no reason to turn to the teachings of Schwarz when making a solid silicon dioxide glass component, as in Kawaguchi. Moreover, in view of the differences between Kawaguchi's silicon dioxide glass components and Schwarz's highly dispersed, pyrogenic

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silicic acid, one of ordinary skill in the art would have no reasons to expect that materials (i.e., polymethylcyclsiloxanes) used in one process would be workable in the other. Indeed, one wishing to make solid silicon dioxide glass components would not expect a technique for making dispersed silicic acid to be useful.

Since Kawaguchi and Schwarz are not properly combinable, the rejection based on that combination should be withdrawn.

The rejection of claims 7, 9-10, 17, and 19-20 under 35 U.S.C. § 103 for obviousness over U.S. Patent No. 3,823,995 to Carpenter ("Carpenter") in view of Kawaguchi and Schwarz is respectfully traversed.

Carpenter is directed to a method for making optical waveguides with silicon tetrachloride.

Kawaguchi and Schwarz are not combinable for the reasons noted above. Carpenter does not overcome this deficiency, because it, like Kawaguchi, makes silicon dioxide articles but without using polymethylcyclsiloxanes. Thus, Carpenter is no more combinable with Schwarz than Kawaguchi. Since the art makes no suggestion of using a polymethylcyclsiloxane for anything other than particulate pyrogenic silicic acid, there would (for the reasons noted above) have been no motivation to combine Kawaguchi or Carpenter with Schwarz, nor any expectation that if such a combination were made (and a polymethylcyclsiloxane starting material were used) a useful solid silicon dioxide mass would result. Since the art fails to teach that such siloxanes are useful in making bodies by soot deposition, the rejection of the claims based on the combination Carpenter, Kawaguchi, and Schwarz should be withdrawn.

Finally, applicants submit that it was improper to make the outstanding office action final. In the first office action, the application was objected to as lacking the assent of all assignees and for having a defective declaration, while claims 28-29 and 31-32 were rejected under 35 U.S.C. § 112. There were no other claims rejected and no art was applied against the claims. In fact, claims 1, 4-23, 26-27, and 30 were deemed allowable over the prior art of record.

In response to the first office action, applicants amended claims 28-29 and 31-32 and submitted a new statement by assignee and a new declaration. This submission in no way justified the PTO's making the above art rejections in the outstanding office action and making that office action final.

The rationale for the finality of the outstanding office action is apparently applicants' submission of a Supplemental Information Disclosure Statement under 37 CFR

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§§ 1.97-1.98. This is plainly not an appropriate basis for making a final rejection. When the present reissue application was filed, applicants submitted an Information Disclosure Statement under 37 CFR §§ 1.97-1.98, citing, *inter alia*, Carpenter, Kawaguchi, Kratel, and Schwarz. Copies of all of these references were provided to the PTO at that time. In addition, applicants' originally-filed Combined Declaration and Power of Attorney specifically mentions Kawaguchi as a basis for the invalidity of certain claims of U.S. Patent No. 5,043,002, which corresponds to the present reissue application. In the first office action, the PTO-1449 form submitted with the Information Disclosure Statement under 37 CFR §§ 1.97-1.98 was only initialed for some of the listed references (e.g., Carpenter) but not others (e.g., Kawaguchi, Kratel, and Schwarz) which were crossed off, because the PTO-1449 form contained no publication date for them. With the response to the first office action, applicants submitted a new PTO-1449 form listing the references that had been previously crossed off. In addition, applicants filed a distinct Supplemental Information Disclosure Statement under 37 CFR §§ 1.97-1.98, together with the fee required pursuant to 37 CFR §§ 1.17(p) and 1.97(c), in order to bring to the PTO's attention 3 additional Japanese patent applications, none of which were applied against the claims in the outstanding office action. Thus, the outstanding office action made rejections of claims which had not been modified in response to the first office action based on 4 newly-applied references which were cited at the outset to the PTO (with one being emphasized in the original declaration). Under such circumstances, there can be no justification for making the outstanding office action final.

The Manual of Patent Examining Procedure § 609(B)(2)(I) provides no support for making a final rejection in the present case. The references submitted with the Supplemental Information Disclosure Statement under 37 CFR §§ 1.97-1.98 were not used to reject the claims in the outstanding office action. The references applied against the claims in the outstanding office action were provided with the original Information Disclosure Statement under 37 CFR §§ 1.97-1.98 and were fully available to the PTO prior to the first office action. The fact that the PTO-1449 form was not fully completed (which is not even the case for Carpenter) is irrelevant.

For all the reasons noted above, the outstanding office action should not have made final.

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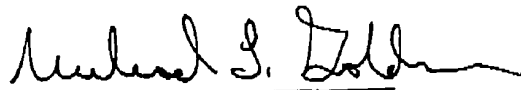
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In view of all of the foregoing, applicants submit that this case is in condition  
for allowance and such allowance is earnestly solicited.

Respectfully submitted,

Date: July 9, 1999



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# HACKH'S CHEMICAL DICTIONARY

[American and British Usage]

*Containing the Words Generally Used in Chemistry,  
and Many of the Terms Used in the Related  
Sciences of Physics, Astrophysics, Mineralogy,  
Pharmacy, Agriculture, Biology,  
Medicine, Engineering, etc.*

*Based on Recent Chemical Literature*

FOURTH EDITION

Completely Revised and Edited by

**JULIUS GRANT**

M.Sc., Ph.D., F.R.I.C. CHEMICAL CONSULTANT

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PAGE 2

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**ENCYCLOPEDIA OF  
CHEMICAL  
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VOLUME 21  
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# SILICA (INTRODUCTION) 993

mission, especially in the ultraviolet. Bulk vitreous silica is difficult to work because of the absence of network-modifying ions present in common glass formulations. An extensive review of the properties and structure of vitreous silica is available (72).

Amorphous silica exists also in a variety of forms that are composed of small particles, possibly aggregated. Commonly encountered products include silica sols, silica gels, precipitated silica, and pyrogenic silica (9,73). These products differ in their modes of manufacture and the way in which the primary particles aggregate (Fig. 8). Amorphous silicas are characterized by small ultimate particle size and high specific surface area. Their surfaces may be substantially anhydrous or may contain silanol,  $-\text{SiOH}$ , groups. These silicas are frequently viewed as condensation polymers of silicic acid,  $\text{Si}(\text{OH})_4$ .

Silica sols are often called colloidal silicas, although other amorphous forms also exhibit colloidal properties owing to high surface areas. Sols are stable dispersions of amorphous silica particles in a liquid, almost always water. Commercial products contain silica particles having diameters of about 3–100 nm, specific surface areas of 50–270  $\text{m}^2/\text{g}$ , and silica contents of 15–50 wt %. These contain small (<1 wt %) amounts of stabilizers, most commonly sodium ions. The

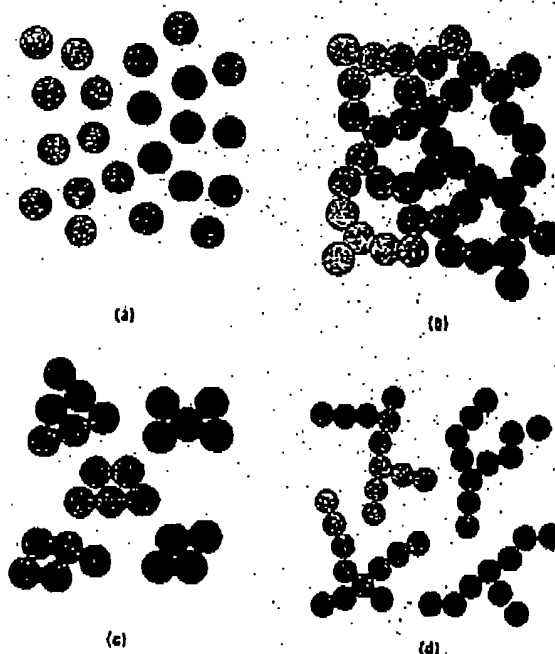


Fig. 8. Different forms of commercially important amorphous silica: (a) sol, (b) gel, (c) precipitate, and (d) pyrogenic.

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# SILANOL

610

**silanol**  $R_3Si(OH)_2$ . See *silane*.  
**silantriol**. A hypothetical hydrolysis product of a monosubstituted chlorosilane of the type  $R-Si(OH)_2$ .  
**Silantriols** condense to form 3-dimensional polymeric resins.  
**silanol**, **Silicool**. The trivalent group  $\equiv SiOH$ .  
**Silastic**. Trademark for a heat-stable silicone, q.v.  
**Cf. silicone rubber**  
**silavans**. Group name for colorless, high-melting-point, strong polymers, containing silicon, carbon, and nitrogen.  
**silbamin**. Silver fluoride.  
**Silberrad**, Oswald John. 1878-1960. British chemist, noted for his work on explosives.  
**Silicite** explosive. A high explosive: potassium chlorate 75, nitrated resin 25%.  
**silix**. A heat- and shock-resistant glass (98% quartz).  
**liquid**. Water glass.  
**Sil-Pos**. Trademark for an alloy, m. 625-705; Cu 80, Ag 16, P 3%; used for brazing alloys containing copper.  
**silica**,  $SiO_2$  = 60.1. Silicon dioxide, silicic acid anhydride. Occurs abundantly in nature (12% of all rocks), and exists in 7 crystalline forms. Classification: (1) Phenocrystalline or vitreous minerals; see *quartz*. (2) Cryptocrystalline and amorphous minerals; see *chalcedony*. (3) Amorphous and colloidal minerals; see *opal*. amorphous. Colorless powder, m. 1650, insoluble in water, soluble in hot alkalies or hydrofluoric acid; used for chemical glassware. colloidal. See *silicic acid*. crystalline. Colorless, transparent prisms, m. 1760, insoluble in water, soluble in hydrofluoric acid. Used in optical instruments, kitchenware, and chemical plant. The main crystalline forms (quartz, tridymite, and cristobalite) have definite transition points (870 and 1470°C, respectively).  
 a. brick. A firebrick containing over 92%  $SiO_2$ ; its crystalline phase is cristobalite and tridymite. s. gel. Gelatinous  $SiO_2$  which, if activated, absorbs water; used to dry blast-furnace gases, air, and other gases. s. minerals. Rock-forming minerals comprising the groups, q.v.: amphiboles, andalusite, calcicite, cordierite, chlorite, feldspar, garnet, ilmenite, leucite, melilite, mica, nephelinite, olivine, pyroxene, scapolite, topaz, tourmaline, zeolite, zirconite; also beryl, quartz, serpentine, talc.  
**silicam**.  $Si(NH_2)_4$  = 100.2. White powder from heating silicon imide. Insoluble in water; forms silicon nitride,  $Si_3N_4$ , when further heated.  
**silicane**. (1) A silane, i.e., a compound of the type  $SiH_4$ . (2)  $SiR_3$ . R is a hydrocarbon radical. (3)  $SiH_3$  = 32.08. Monosilane, silicomethane, silico-hydride. Colorless gas, b. -112. bromo-  $SiH_3Br$  = 111.0. Colorless gas, b. -1.8. chloro-  $SiH_3Cl$  = 86.64. Colorless gas, b. -30. di-  $SiH_2$  = 62.16. Silicoethane, a gas, m. -132. dibromo-  $SiH_2Br_2$  = 189.91. Colorless liquid, d. 2.17, b. 66. dichloro-  $SiH_2Cl_2$  = 100.99. Colorless gas, b. 3.3. dimethyl-  $SiH_2Me_2$  = 60.12. Dimethylmonosilane. Colorless gas, b. -20. ether-  $(SiH_3)_2O$  = 78.17. Disilane oxide. Colorless gas, b. 15. ethoxytriethyl-  $Et_3SiOEt$  = 160.22. Triethylsilane ethyl ether, triethyl silico ethyl ether. Colorless liquid, b. 153, insoluble in water. hydroxy- **Silicool**, methyl-  $MeSiH_3$  = 46.11. Methylmonosilane. Colorless gas, b. -57. tetra-  $SiH_4$  = 122.31. Silicoethane. A gas, m. -94. tetrabromo- Silicon bromide.

# SILICO-

tetrachloro- Silicon chloride. tetraethyl-  $SiEt_4$  = 144.25. Silicon tetraethyl, silicomonane. Colorless liquid, d. 0.7083, b. 153. tetrachloro- Silicon fluoride. tetrachloro- Silicon iodide. tetramethyl-  $SiMe_4$  = 144.22. Silicon tetramethyl. Colorless liquid, d. 0.648, b. 27. tetraphenyl-  $Si(C_6H_5)_4$  = 236.24. Silicon tetraphenyl, tetraphenyl silicon. Colorless crystals, m. 333. tri-  $Si_3H_8$  = 92.24. Silicopropane. A gas, m. -117. tribromo-  $SiH_3Br_3$  = 366.62. Silicobromopropane. Colorless liquid, d. 3.7, b. 109. trichloro- Silicochloroform. trichloroethyl-  $Si(C_2H_5)Cl_3$  = 183.47. Colorless liquid, d. 1.339. trichlorophenyl-  $Si(C_6H_5)Cl_3$  = 211.47. Colorless liquid, d. 1.338, b. 137, decomp. in water. triethyl-  $(C_2H_5)_3SiH$  = 116.18. Triethyl silicon, silicoheptane. Colorless liquid, d. 0.761, b. 107, insoluble in water. trifluoro-  $SiH_3F_3$  = 86.07. Silicofluoroform. Colorless gas, b. -80. trilocho-  $Si_3H_8$  = 409.63. Silicoiodoform. Red liquid, d. 3.314, b. 320.  
**silicate**. A salt derived from silica or the silicic acid. Silicates form the largest group of minerals (see *silica*), and are derived from  $H_2SiO_4$ , orthosilicic acid, and  $M_2SiO_4$ , metasilicic acid, which may combine to form polysilicates. Except for the alkali silicates, they are insoluble in water. See *silica minerals*. fibrous- Natural: Asbestos. Man-made: Glass, silica, and aluminosilicate fibers, rock wool, slag wool.  
 s. cement. See *silica cement*. s. of soda. Sodium silicate.  
**siliceous**. Containing silica. s. algae. See *algae*. s. deposit. S. sinter. The solid accumulation of silica deposited from hot mineral springs. Cf. *geyserite*. s. earth. Silice of diatomite origin, purified by boiling with dilute acid, washing, and calcining; a filtermedium (U.S.P.). s. sinter. S. deposit.  
**silicic**. (1) Containing silicic acid. (2) Containing silicic acid. s. acid.  $H_2SiO_4$  = 66.2. Orthosilicic acid. White powder, slightly soluble in water. di-  $H_2SiO_3$  or  $H_2SiO_4$ . White, insoluble powder, meta-  $H_2SiO_3$  = 78.1. White powder, insoluble in water. tri-  $H_2SiO_3$  = 216.3. White, insoluble powder.

# -- SILICIC ACIDS

$H_2SiO_4$  = 48.0.  $H_2O$ , tetra-  
 $H_2SiO_3$  = 28.0.  $H_2O$ , meta-di-  
 $H_2SiO_2$  = 28.0.  $H_2O$ , meta-tri-  
 $H_2SiO_2$  = 28.0.  $H_2O$ , meta-  
 $H_2SiO_2$  = 28.0.  $H_2O$ , ortho-tri-  
 $H_2SiO_2$  = 28.0.  $H_2O$ , ortho-di-  
 $H_2SiO_2$  = 28.0.  $H_2O$ , ortho-

**silicids**.  $M_2Si$ . M is Fe, Ni, Co, Cr, Mn, Cu, or Mg.  
**silicification**. The gradual replacement of rocks or fossils by silica (petrification).  
**silicified**. Describing an organic material, e.g., wood, that has been petrified.  
**silicium**. Silicon.  
**silico-** Prefix indicating silicon, generally in organic compounds. s. benzoic acid.  $PhSiOOH$  = 135.1. m. 92, insoluble in water. s. bromoform.  $SiHBr_3$  = 268.6. Heavy, colorless liquid, d. 2.7, b. 116, decomp. by water. s. butane. See *silanes*. s. calcium. A product of the electric furnace used to deoxidize steel. s. chloroform.  $SiHCl_3$  = 185.26

# SILICOL

Colorless liquid, d. 1.3  
 a. decitungstic acid.  
 powder; a reagent for  
 s. ethane. See *silanes*  
 s. fluoric acid. Fluosil  
 leucine. s. heptane.  
 silane. Colorless liquid  
 Silanes. s. iodoform.  
 colorless liquid, d. 3.4  
 s. methane. Silane. s.  
 = 122.2. White, unsl.  
 S. decitungstic acid.  
**silicol**.  $R_2SiOH$ .  
 $Et_2SiOH$  = 132.18. S.  
 less liquid, b. 154, insol.  
**silicon**.  $Si$  = 28.09. Silici  
 of the carbon group.  
 modifications: (1) Am  
 d. 3.35. (2) Crystalline,  
 insoluble in water.  
 crystals, or graphite-like  
 molten. (4) Adamant  
 pal valency 4. S. forms  
 in the earth surface (r  
 impart hardness, and  
 minerals. ethyl- The ra  
 methyl- The radical  $\equiv S$   
 mass 27. Cf. *radiocline*.  
 s. alkyls. (1) Hydrog  
 spreading with hydrocarb  
 Organic compounds of s  
 $SiMe_4$ . See *silanes*.  
 alloys of s. with metals: a  
 s. borides.  $SiB$ ,  $SiH$ ,  
 irregular crystals, of high  
 conductors of electricity.  
 347.9. S. tetrabromide.  
 b. 154, decomp. by wat  
 $Si_2Br_6$  = 535.7. S. trib  
 b. 240, decomp. by wat  
 corrodible alloy: Cu, Sn, w  
 $SiC$  = 40.1. Colorless  
 used in refractories and  
 (1)  $SiCl_4$  = 170.0. S. t  
 fuming liquid, d. 1.534, b. 5  
 silicic acid. Used in elec  
 with ammonia vapors, i  
 $Si_2Cl_6$  = 569.0. S. trichlo  
 water. (3)  $Si_2Cl_6$  = 3  
 White powder; s. copper  
 Cu 70-80%. used in n  
 silica. s. disulfide,  $SiS_2$ ,  
 sublime when heated, decomp  
 See *silanes*. s. ethyl. 1  
**silicose**. s. fluorides. (1)  
 tetrafluoride. Colorless, su  
 -65, decomp. by water to  
 in alcohol. (2)  $Si_2F_6$  =  
 White powder. s. hydri  
 iodides. (1)  $SiH_4$  = 535.8.  
 less solid, m. 121, insoluble  
 817.7. S. subiodide. Colo  
 vacuo, decomp. by water.  
 Iron containing 2-15% Si;  
 s. magnesium.  $Mg_2Si$  = 76.7  
 Gray leadlike, decomp. by  
 methane. Silane. s. meth  
 Colorless liquid, b. 26. s. nit

Exhibit 1 (cont.)

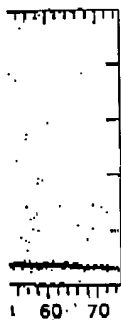
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bases. These methods are qualitative, however, and fail to satisfy regulatory requirements to determine crystallinity at 0.1% concentration in bulk materials.

Microamorphous silica can be divided into microparticulate silica, ie, microscopic sheets and fibers, and highly hydrated silica (1). The microparticulate silicas are the most important group commercially. These include silicas precipitated from aqueous solution and silicas formed in the vapor phase, called pyrogenic or fumed silica. Several synthetic routes exist to prepare any form of microporous silica, where microporous often refers to the ability of the silica to adsorb gases or liquids. For example, pyrogenic silica can be prepared by either (1) vaporizing silicon dioxide in an arc or in a plasma jet, and condensing it in an inert gas; (2) oxidizing the more volatile silicon monoxide, SiO, in the vapor phase with air, and condensing it; or (3) oxidizing silicon compounds (qv), such as SiH<sub>4</sub>, SiCl<sub>4</sub>, or HSiCl<sub>3</sub>, in the vapor phase in a hydrocarbon flame, and then condensing to produce branched-chain aggregates of silica up to approximately 1  $\mu$ m in length (Fig. 6). Pyrogenic silica is collected as a finely divided powder.

Amorphous silica formed in aqueous solution can occur as sols, gels, or particles. A silica sol can either consist of polysilicic acid having a molecular weight of SiO<sub>2</sub> up to about 100,000, or be a stable dispersion of fine colloidal particles of diameters >5 nm (Fig. 7). A silica gel has a three-dimensional, continuous structure (Fig. 8). Macroscopic particles formed by aggregation of

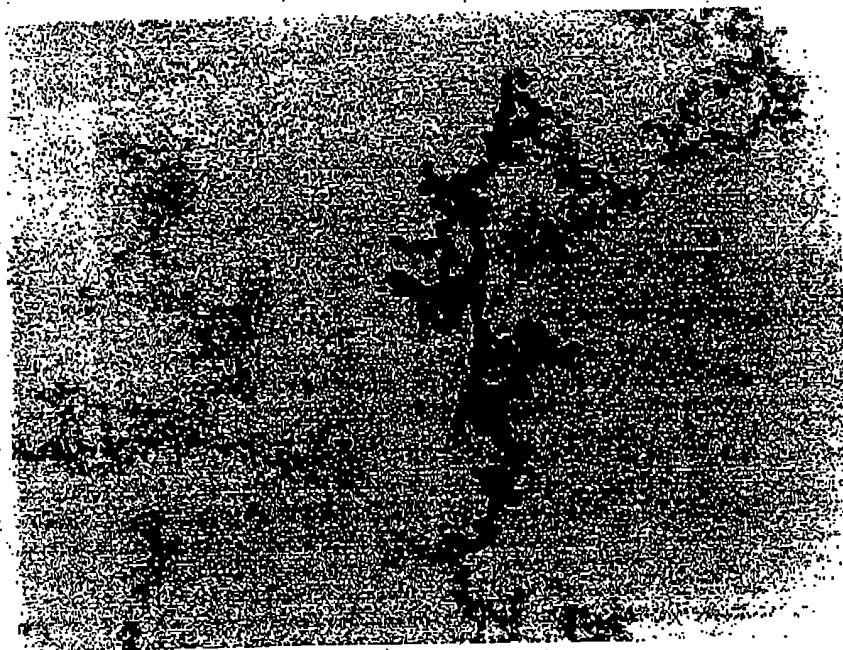


Fig. 6. Transmission electron micrograph of a commercial pyrogenic silica, Wacker HDK. Magnification of 225,000 $\times$ .

Exhibit 2 (cont.)

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